PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : G01B 9/02

A1

(11) International Publication Number: WO 99/64814

(43) International Publication Date: 16 December 1999 (16.12.99)

(21) International Application Number: PCT/US99/13339

(22) International Filing Date: 11 June 1999 (11.06.99)

(30) Priority Data:

60/089,089	12 June 1998 (12.06.98)	US
09/329,520	10 June 1999 (10.06.99)	US

(81) Designated States: CA, IL, JP, SG, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published

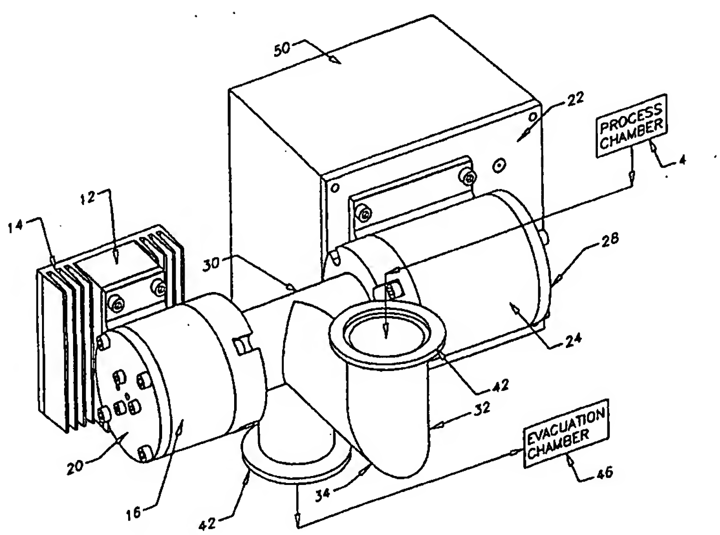
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(71) Applicant: ON-LINE TECHNOLOGIES, INC. [US/US]; 89 Church Street, P.O. Box 380379, East Hartford, CT 6138-0379 (US).

(72) Inventors: SPARTZ, Martin, L.; 29 Arril Drive, East Windsor, CT 06088 (US). BONANNO, Anthony, S.; 11 Scott Lane, Windsor, CT 06095 (US). ROSENTHAL, Peter, A.; 10 Farmstead Lane, West Simsbury, CT 06107 (US). RICHTER, Matthew; 1409 East San Antonio Street, San Jose, CA 95116 (US).

(74) Agent: DORMAN, Ira, S.; Suite 200, 330 Roberts Street, East Hartford, CT 06108 (US).

(54) Title: METHOD AND APPARATUS FOR DETERMINING PROCESSING CHAMBER CLEANING OR WAFER ETCHING ENDPOINT



(57) Abstract

The gas withdrawn from a silicon wafer processing chamber, during wafer etching or chamber cleaning with a fluoride free radical-containing plasma, is analyzed optically. The apparatus consists of an infrared radiation source (10) mounted in a holder (12), to which is attached a heat sink (14). The holder (12) is mounted upon a source mirror housing (16), which contains a collimating mirror (18) supported on a source mirror holder (20). The filter sensor unit includes an endpoint instrument assembly mounting plate (22), which is mounted upon a detector mirror housing (24) and in turn contains detector focussing mirror (26) supported upon a detector mirror holder (28).

METHOD AND APPARATUS FOR DETERMINING PROCESSING CHAMBER CLEANING OR WAFER ETCHING ENDPOINT

CROSS-REFERENCE TO RELATED PROVISIONAL APPLICATION

This application claims the benefit of provisional Application No. 60/089,089, bearing the foregoing title and filed on June 12, 1998 in the name of Martin L. Spartz, one of the inventors designated herein.

BACKGROUND OF THE INVENTION

During silicon wafer processing (i.e., fabrication or etching) the processing chamber becomes contaminated with silicon materials (as well as photoresist materials, metals, metal oxides, and dielectrics), which must be removed after a predetermined or suitable number of wafers have been produced. It is important not only that thorough cleaning be achieved, but also that the cleaning operation be completed as quickly as possible so as to minimize toxic emissions and chamber degradation, and also to avoid the excessive consumption of gaseous cleaning agents.

In accordance with current practice, a plasma containing fluorine free radicals (F·) is struck in the processing chamber to effect cleaning; the free radicals react with the contaminating silicon material to form various volatile species, including SiF_4. Optical sensors are used to monitor the emission intensity of the excited unreacted fluorine free radicals remaining in the plasma, to thereby determine the cleaning end point.

Similar plasma processes are used to etch silicon and silicon-containing layers on the wafers themselves, as part of the wafer-manufacturing process. Optical emission sensors are commonly used to determine endpoints of these processes, as well. (As used herein, references to wafers can generally be construed more broadly, as encompassing articles and devices comprised of silicon.)

Manufactures of wafer fabrication tools are now developing procedures in which the chamber cleaning or wafer etching plasma is struck at a remote location so as to reduce wear and other adverse effects upon the processing chamber, and to modify the kinetics of the wafer etch process. This of course makes it

unfeasible to determine the cleaning end point by measuring optical emission from the chamber.

SUMMARY OF THE INVENTION

Accordingly, the broad objects of the present invention are to provide an apparatus and method for quickly, effectively, and accurately determining the end point or other parameters of a process carried out at a reaction site and, in particular, the end point of etching or of processing chamber cleaning in connection with the fabrication of semiconductor wafers and devices.

More specific objects of the invention are to provide such an apparatus and method especially for the etching of silicon wafers and the cleaning of chambers in which they are processed, whereby the cleaning end point or other parameter is determined by analysis of the gaseous product produced in the processing chamber and, in particular, by optical analysis of the gas for the virtual absence, or significant change in the concentration, of a silicon reaction product indicator species.

It has now been found that certain of the foregoing and related objects of the invention are broadly attained by the provision of an optical method for the detection of the endpoint of a process that is carried out at a reaction site, in which process substances react chemically to produce an analyte gas containing a volatile chemical indicator species having a radiation absorption characteristic, indicative of the concentration of said indicator species in said analyte gas, at at least a first wavelength. The method comprises the steps:

(a) projecting at least one beam of radiation through the analyte gas at a sampling location (which may be the reaction site or, more desirably in many instances, a remote location to which the analyte gas is withdrawn), the beam including at least the "first" wavelength;

(b) detecting radiation of the first wavelength that passes through the analyte gas, and detecting radiation of the "at least

one" beam that passes through the analyte gas substantially unattenuated;

(c) generating first and second electrical signals that are indicative, respectively, of the intensity of radiation of the first wavelength and of the unattenuated beam;

(d) analyzing the electrical signals to determine the level of absorption of the first wavelength of radiation, by the indicator species, that occurs during passage of the beam through the analyte gas, and thereby to provide an indication of the concentration of the indicator species therein; and

(e) relating the concentration of the indicator species to a parameter of the process (which phrase is intended to be inclusive of the properties of any product of the process), generally by performing experiments to relate the concentration of the indicator species to the desired property or parameter of the process.

In carrying out the method the "second" signal (a reference signal) may conveniently and effectively be obtained by: (1) detecting, effectively separately from the first wavelength, the intensity of radiation of a second wavelength, projected through the analyte gas, for which the analyte gas does not have a significant absorption characteristic; (2) detecting, effectively separately from the first wavelength, the intensity of radiation of a spectral beam, projected through the analyte gas, wherein the spectral beam contains a range of frequencies to the absorption of which, by the analyte gas, the indicator species makes no more than a minor contribution; or (3) detecting radiation of at least the first wavelength projected through a modified form of the analyte gas, the modified form differing effectively from the analyte gas itself only by being substantially devoid of the indicator species.

Generally, the at least one beam (which will usually be a single beam) will be comprised of discrete infrared spectral regions including such first and second wavelengths, and will be filtered optically prior to detection to discriminate the wavelengths from one another. When the reaction site is a chamber for fabrication of silicon semiconductor devices, the substances

that react with one another will include silicon and usually a fluorine species, with the analyte gas preferably comprising a fluorine-containing plasma or a fluorine-containing plasma product; the indicator species will preferably be SiF_4, and the first wavelength will have a nominal value of 9.7 microns.

In specific applications, the method will additionally include the preliminary steps, effected in the processing chamber, of (f) etching silicon from at least one device comprised of silicon using a fluorine plasma, the method being carried out for the purpose of determining the end point of a process of either cleaning of surfaces within the chamber or of etching of the silicon device. The method may additionally include the steps, effected prior to the foregoing step (a) and advantageously by use of Fourier Transform Infrared (FT-IR) spectroscopy, filter-based spectroscopy, and dispersive spectroscopy, of:

(g) projecting a beam of spectral radiation through the analyte gas at the sampling location;

(h) analyzing the beam of spectral radiation after passage through the analyte gas to identify at least one wavelength of radiation that is strongly absorbed; and

(i) carrying out such further steps as may be necessary to establish a qualitative and quantitative correlation between the absorption of the at least one wavelength and the concentration of the indicator species in the analyte gas, to thereby establish the "first" wavelength for use in subsequent steps of the method.

In certain embodiments, the method of the invention is employed for the detection of an etch rate, deposition rate, etch amount, deposition amount, and/or faults that are achieved or occur in a process, utilizing the steps herein set forth.

Other objects are attained by the provision of apparatus for measuring a volatile chemical species that is generated at a reaction site and is contained in an analyte gas withdrawn therefrom, the apparatus comprising:

(a) means for generating at least one beam of radiation containing at least a first wavelength, preferably in the infrared

range, that is absorbed strongly by the generated chemical species that is to be measured;

(b) a conduit for gas flow from the reaction site, the conduit constituting a sampling site and having windows fabricated of a composition that is resistant to corrosion by the analyte gas, that at least limits process contamination, and that is transparent to the at least one beam, the windows being aligned for effective traversal of the analyte gas, by the generated radiation beam, in the gas flow conduit;

(c) means for directing the radiation beam for entry through one of the windows of the gas flow conduit;

(d) at least one radiation detector that is responsive to at least the first wavelength of radiation, and that is constructed for generating a first electrical signal that is indicative of the intensity of at least the first wavelength of radiation, the detector being operatively disposed to responsively intercept the at least one radiation beam exiting the gas flow conduit through the other of the windows;

(e) means for generating a second signal that is indicative of the intensity of radiation of the "at least one" beam that passes through the analyte gas substantially unattenuated by absorption of radiation by the indicator species; and

(f) signal interpretation means, including electronic data processing means, for analyzing the first and second electrical signals to determine the level of absorption of at least the first wavelength of radiation, by the indicator species, that occurs during passage of the beam through the analyte gas.

In preferred embodiments of the apparatus the means for generating (and modulating) the beam will comprise a Fourier Transform Infrared spectrometer. The apparatus may comprise separate means, such as a filter wheel, operatively disposed in the beam path for modulating the beam of radiation so as to discriminate radiation of different wavelengths, and most desirably the reaction site will comprise a processing chamber for fabrication of silicon semiconductor devices.

As will be appreciated, in carrying out the method and utilizing the apparatus zero absorption, or absorption below

a specified level, of the efficiently absorbed wavelength of radiation will be indicative of the absence, or of the attainment of a value below a threshold level, of the chemical species generated by reaction with the contaminating silicon material. That will in turn indicate that the endpoint for cleaning of the processing chamber or for etching of the wafer or other device, has been attained. A correlation between the indicator species and such an end point (or to etch or deposition rates or amounts, or process faults, as the case may be), can be established empirically or by other means that will be evident to those skilled in the art. Although the sampling location will desirably be remote from the reaction site, that need not be so; i.e., sampling may occur in situ, by projecting the radiation beam through the processing chamber itself.

The radiation utilized for the species measurement will usually lie in the infrared spectral region, typically being generated by a hot body globar, a diode laser, or an LED. A Fourier Transform Infrared spectrometer may be employed to generate and modulate radiation in the selected spectral ranges, or a filter wheel, a tunable laser, a grating, or a prism absorption cell, coupled with a chopper wheel, may be utilized for that purpose. Focusing mirrors and/or lenses will normally be used for collecting the radiation and directing it along the beam path through the conduit and upon the detector. Suitable materials for fabrication of the corrosion-resistant windows include calcium fluoride, potassium bromide, potassium fluoride, and (preferably) barium fluoride, and although a $LiTaO_3$ detector will generally be utilized other detectors, such as MCT, lead salt, and DTGS devices, may be employed as appropriate and as may be desired.

Signal interpretation may be carried out through classical least squares quantitation routines using, as the basis for comparison, nitrogen, vacuum, or nonabsorbing gas in the conduit; a library of spectra; or calibration runs. The signal processing means will comprise a logic circuit, data storage capacity, and appropriate electronics, and may for example take the form of an amplifier, a digitizer, and a computer programmed to carry

out the necessary digital computations. In the preferred embodiments the apparatus will be configured to detect SiF_4 in the analyte gas, that product being one of the species that are formed by the reaction of fluorine free radicals (present in the cleaning plasma) with the silicon material contaminant, and being characterized by strong absorption of radiation at 9.724μm.

BRIEF DESCRIPTION OF THE DRAWING

Figure 1 is a perspective view showing apparatus embodying the present invention, with associated components of a wafer processing tool; and

Figure 2 is an exploded perspective view, drawn to a reduced scale, of the apparatus of Figure 1.

DETAILED DESCRIPTION OF THE PREFERRED AND ILLUSTRATED EMBODIMENTS

The illustrated apparatus consists of an infrared radiation source 10 (a globar) mounted in a holder 12, to which is attached a heat sink 14 (which is optional). The holder 12 is mounted upon a source mirror housing 16, which contains a collimating mirror 18 supported on a source mirror holder 20. The filter sensor unit includes an endpoint instrument assembly mounting plate 22, which is mounted upon a detector mirror housing 24 and in turn contains a detector focussing mirror 26 supported upon a detector mirror holder 28. The housings 16, 24 are attached to a transverse section 30 formed on an offset conduit 32 of a vacuum flange, generally designated by the numeral 34, and the mirrors 18, 26 are aligned for optical communication through BaF_2 windows 36 provided at opposite ends of the transverse section 30; the windows 36 are held in place by retaining rings 38, and are sealed by VITON o-rings 40. KF40 connections 42, at the inlet and outlet ends of the conduit 32, enable attachment of the vacuum flange 34 to a wafer processing chamber 44 and an evacuation system 46, respectively. An end point instrument assembly 48 (shown diagrammatically) is supported on the mounting plate 22 and is enclosed within the cover 50.

As will be appreciated, a beam of radiation B generated by the IR source 10 is collimated by the mirror 18 in the housing

16 and is projected through the transverse section 30 of the vacuum flange 34 and the BaF_2 windows 36 at the opposite ends thereof. The beam impinges upon the mirror 26 in the housing 24, and is focussed thereby upon a detector of the instrument assembly 48, passing through appropriate filters (as will be described more fully below) of which the assembly 48 is also comprised. The intensity of radiation sensed by the detector will of course be attenuated by any absorbing molecules contained within the gas stream flowing through the offset conduit 32 of the vacuum flange 34 from the processing chamber 44, under the influence of the vacuum system 46.

In a specific example, an infrared filter-based instrument measures the concentration of SiF_4 produced during cleaning of the chamber of a high density plasma, chemical vapor deposition silicon wafer processing system. Unique to the present invention is the recognition that SiF_4 serves as a highly effective and definitive indicator of the clean or etch end point; i.e., when the SiF_4 concentration decreases to an undetectable or threshold level the cleaning or etching end point is deemed to have been reached.

More particularly, to carry out the analysis a two-filter IR sensor, providing two spectral band passes, is employed. The center wavelength for one of the filters is selected to lie at 9.724μm, which corresponds to a wavelength of strong SiF_4 absorption; the other filter functions at 9.091μm, which corresponds to no significant absorption from the process gas. The unit includes three major parts: an IR source assembly, a vacuum gas cell that mounts into the processing chamber vacuum duct, and the IR sensor that determines continuously the SiF_4 concentration during the chamber clean or wafer etch; a globar provides an active (hot) IR source, which allows specie detection without plasma emission.

It is of course known that almost all molecules absorb radiation in some portion of the infrared spectrum, from 1 to 200μm in wavelength (10,000 to 50 wavenumbers per centimeter). Radiation is absorbed by any given molecule at frequencies that match the frequencies at which the molecule vibrates, which in

turn depends upon the mass and electronic structure of the constituent atoms and is attributable primarily to stretching and bending motions; most molecules have many bonds and absorb at many different frequencies.

From experiments performed using an FT-IR spectrometer, SiF_4 has unexpectedly been found to be perhaps the best indicator of the state of cleaning of the processing chamber; it absorbs light at 1028 cm^{-1} (9.714μm) in direct proportion to its concentration. From the data collected it is anticipated that, if chamber cleaning is effected after each wafer is processed, the maximum percentage of light absorbed will be about 50% for the various doped and undoped silicon glasses. The current detection limit of the sensor, using a 7.925cm beam path, is about 3.0 mtorr; this corresponds to about 3.7 μmol, or about 2.2 x 10^{18} molecules, of SiF_4 in the IR beam.

In one exemplary embodiment, filter spectrometry is employed to carry out the method of the invention. The filters used for the detection of SiF_4 are desirably found to have the following characteristics:

Analyte Filter:	
Center Wavelength	9.724μm ± 0.100μm
Half Peak Wavelength	0.225μm ± 0.025μm
Peak Transmittance	65% minimum
Out of Band Transmittance	blocked UV through 18μm
Reference Filter:	
Center Wavelength	9.091μm ± 0.100μm
Half Peak Wavelength	0.225μm ± 0.025μm
Peak Transmittance	65% minimum
Out of Band Transmittance	blocked UV through 18μm
Out of Band Filter:	
Out of Band Transmittance	blocked at λ<8μm and >14μm
Filter Substrates:	
Width	0.605" ± 0.005"

Height	0.320" ± 0.005"
Thickness	1.0 mm ± 0.1 mm
Surface Quality	80:50 per face
Clear Aperture	0.585 x 0.300"
Operating Temperature	30°C ± 5°C

Most mid-IR sources are hot, DC-powered glowers made from ceramic or metal alloy materials, which generally operate between 900° and 1500°C and (depending upon the size and temperature of the source) may require no cooling. One suitable source is a miniature tungsten carbide glower, operated at 10 volts and 1.8 amps and generating a surface temperature of between 1100° and 1200°C.

The IR light may be collimated to increase light intensity passing through the sample and impinging upon the detector. A suitable front surface mirror design uses a one-inch, 90° off-axis aluminum parabola having an aluminum/MgF_2 surface coating; the IR source is placed at the focal point of the mirror for collimation.

As previously described, the IR source unit will usually be connected to one side of a conduit from the processing chamber. Optical transmission will most advantageously be accomplished using two 4-5 mm thick 25.4 mm BaF_2 windows, mounted on both sides of the flange; the base IR path length through the flange will typically be 3.12 inches.

In such an embodiment the radiation detector unit will be mounted to the adjacent side of the flange to complete the detection system. The IR light will be focussed, using a front surface mirror, and a mirror matching the source mirror will focus light into the detection housing. Modulation of the IR light is effected using a chopper wheel measuring about 1.5 inch in diameter and spinning at 5 Hz, which wheel contains the two required optical filters (analyte and reference), as described. Each filter allows transmission during approximately 25% of the chopper rotation cycle, with transmission being blocked during the remaining 50% of the cycle to achieve optimal modulation; the arrangement produces and on-off signal twice per rotation, one signal

being the reference signal and the other being the analyte signal. The light then passes through a third filter to assure no out-of-band transmission of the IR radiation.

A lithium tantalate ($LiTaO_3$) pyroelectric IR detector is employed, which is sensitive to thermal energy and is room temperature-compensated to correct for thermal drift. As the detector temperature changes (due to varying light intensity), corresponding increases and decreases of polarization occur on the dielectric material, which in turn produce variations in charge flow. The detector functions most effectively at slower modulation frequencies, generally less than 10 Hz, and its window is designed to pass 80% of the IR light between 8μm and 14μm.

The two signal amplitudes are collected by a lock-and-hold amplifier. The analyte signal is divided into the reference signal, and a $\log_{10}$ value is computed on the quotient to produce the absorbance output signal. An absorbance value of 0.300 abs produces an output of 5 V above the baseline signal; as noted above, absorbance is approximately linearly proportional to the gas concentration. The entry of SiF_4 into the beam attenuates the analyte signal, resulting in the generation of a positive voltage response.

In particularly preferred embodiments two outputs, with zero absorbance generating 0.1 and 1 V signals, will be employed. For one signal 5 V will be equivalent to 0.300 abs (about 380 mtorr SiF_4); the other signal will have a value ten times as great, i.e., 5 V will be equivalent to 0.030 abs (about 38 mtorr SiF_4). It is anticipated that the second signal will be driven to a 10 volt maximum when a high level of SiF_4 is present, and that once the concentration decreases below about 68 mtorr a signal having a different value will be observed. Other signals, such as an integrator, may be added for summation of total SiF_4 removed, if so desired.

As noted previously, techniques and mechanisms, other than those that involve the use of discrete radiation regions containing highly absorbed and substantially non-absorbed wavelengths, respectively, can be used to obtain discrete analyte and reference signals. For example, a single optical filter can advantageously

be used to collect the intensity value for a reference signal at a time when the indicator species is known to be absent from the beam path. Alternatively, the reference signal may be obtained from a spectral beam that contains wavelengths which are mostly not absorbed by components of the analyte gas thereby rendering insignificant the intensity contribution to the signal that is made by the highly absorbed "first" wavelength. It will be appreciated that reference herein to etching of silicon, and to the removal of silicon deposits, are intended to encompass compounds of silicon as well, provided of course that an indicator species is produced by reaction of the silicon with the etchant or cleaning agent (especially a fluorine-containing plasma or plasma product).

Replacement of the sensor system described herein is facile, and may require little more than the removal of two flange clamps and disconnection of one or two electrical connections. A replacement sensor system can be ready for use after 5 to 10 minutes of thermal stabilization; it will have the same response to SiF_4 as the original, ensuring similar output from system-to-system.

Thus, it can be seen that the present invention provides an apparatus and method for quickly, effectively, and accurately determining the end point, and other parameters and/or faults, of a process carried out at a reaction site and, in particular, the end point of etching of semiconductor wafers and devices comprised of silicon, and the cleaning of chambers used for processing of such wafers and articles. In either case the end point or other parameter or fault is determined by analysis of the gaseous effluent from the processing chamber, i.e., by optical analysis of the gas for the virtual absence, or significant change in concentration, of a silicon reaction product indicator species.

While the foregoing discussion has pertained predominantly to end point detection, it will be appreciated that quantitative concentration measurements of analyte species can be employed, for example, to determine rates of deposition or etch, given knowledge of the mass flow. Rates can also be integrated over time to determine total quantities etched or deposited. It will also be appreciated that historical quantitative knowledge of

indicator species concentrations may be employed for fault detection and fault classification.

CLAIMS

Having thus described the invention, what is CLAIMED is:

1. An optical method for the detection of the endpoint of a process carried out at a reaction site, in which process substances react chemically to produce an analyte gas containing a volatile chemical indicator species having a significant radiation absorption characteristic, indicative of the concentration of said indicator species in said analyte gas, at at least a first wavelength in the infrared range, comprising the steps:

(a) projecting at least one beam of radiation through said analyte gas at a sampling location, said beam including at least said first wavelength;

(b) detecting radiation of said first wavelength that passes through said analyte gas, and detecting radiation in said at least one beam that passes through said analyte gas substantially unattenuated;

(c) generating first and second electrical signals that are indicative, respectively, of the intensity of radiation of said first wavelength and of said unattenuated beam; and

(d) analyzing said electrical signals to determine the level of absorption of said first wavelength of radiation, by said indicator species, that occurs during passage of said beam through said analyte gas, and thereby to provide an indication of said concentration of said indicator species therein; and

(e) relating said concentration of indicator species to a parameter of said process.

2. The method of Claim 1 wherein said second signal is obtained by a technique selected from the class by consisting of: (1) detecting, effectively separately from said first wavelength, a second wavelength of radiation, projected through said analyte gas, for which said analyte gas does not have a significant absorption characteristic; (2) detecting, effectively separately from said first wavelength, radiation of a spectral beam, projected through said analyte gas, said spectral beam containing a range of frequencies to the absorption of which by said analyte gas said indicator species makes no more than a minor contribution; and (3) detecting radiation of at least said first wavelength projected through a modified form of said analyte gas, said modified form differing effectively from said analyte gas by being substantially devoid of said indicator species.

3. The method of Claim 2 wherein said at least one beam is comprised of said first and second wavelengths of radiation, and is filtered optically prior to said detection to discriminate said wavelengths from one another.

4. The method of Claim 3 wherein said first and second wavelengths are contained in a single beam projected through said analyte gas.

5. The method of Claim 1 wherein said substances that react with one another include silicon and fluorine species.

6. The method of Claim 5 wherein said indicator species consists essentially of SiF_4, and wherein said first wavelength has a nominal value of 9.7 microns.

7. The method of Claim 6 wherein said analyte gas comprises at least one of a fluorine containing plasma and a fluorine containing plasma product.

8. The method of Claim 5 wherein said reaction site is a chamber for fabrication of silicon semiconductor devices.

9. The method of Claim 8 additionally including the step, effected in said chamber and prior to said step (a), of: (f) etching silicon from at least one device comprised of silicon using a fluorine-containing plasma, said method being carried out to determine the end point of a process selected from the class consisting of cleaning of surfaces within said chamber and etching of said at least one device.

10. The method of Claim 1 additionally including the steps, effected prior to said step (a), of:

(g) projecting a beam of spectral radiation through said analyte gas at said remote location;

(h) analyzing said beam of spectral radiation after passage through said analyte gas to identify at least one wavelength of radiation that is strongly absorbed; and

(i) carrying out such further steps as may be necessary to establish a qualitative and quantitative correlation between the absorption of said at least one wavelength and the concentration of said indicator species in said analyte gas, said one wavelength, exhibiting said correlation, constituting said first wavelength employed in subsequent steps of said method.

11. The method of Claim 10 wherein said steps (g) and (h) are effected by a technique selected from the class consisting of Fourier Transform Infrared spectroscopy, filter-based spectroscopy, laser spectroscopy, and dispersive spectroscopy.

12. The method of Claim 1 wherein step (e) is accomplished by performing experiments to relate the concentration of the indicator species to the desired property or parameter of the process.

13. The method of Claim 1 including the additional step, effect prior to said step (a), of withdrawing said analyte gas from said reaction site to a remote location at which said step (a) is carried out, said remote location constituting said sampling location.

14. An optical method for the detection of the etch rate, deposition rate, etch amount, deposition amount, or process fault of a process carried out at a reaction site, in which process substances react chemically to produce an analyte gas containing a volatile chemical indicator species having a radiation absorption characteristic, indicative of the concentration of said indicator species in said analyte gas, at at least a first wavelength in the infrared range, comprising the steps:

(a) projecting at least one beam of radiation through said analyte gas at a sampling location, said beam including at least said first wavelength;

(b) detecting radiation of said first wavelength that passes through said analyte gas, and detecting radiation in said at least one beam that passes through said analyte gas substantially unattenuated by absorption of radiation by said analyte gas;

(c) generating first and second electrical signals that are indicative, respectively, of the intensity of radiation of said first wavelength and of said unattenuated beam;

(d) analyzing said electrical signals to determine the level of absorption of said first wavelength of radiation, by said indicator species, that occurs during passage of said beam through said analyte gas, and thereby to provide an indication of said concentration of said indicator species therein; and

(e) relating said concentration of indicator species to a parameter of said process.

15. Apparatus for measuring a volatile chemical species that is generated at a reaction site and is contained in an analyte gas withdrawn therefrom, the apparatus comprising:

a) means for generating at least one beam of radiation containing at least a first wavelength that is absorbed signifi-

cantly by the generated chemical species that is to be measured;

(b) a conduit for gas flow from said reaction site, the conduit constituting a sampling site and having windows fabricated of a composition that is resistant to corrosion by the analyte gas, that at least limits process contamination, and that is transparent to said at least one beam, said windows being aligned for effective traversal of the analyte gas, by the generated radiation beam, in the gas flow conduit;

(c) means for directing the radiation beam for entry through one of the windows of the gas flow conduit;

(d) at least one radiation detector that is responsive to at least said first wavelength of radiation, and that is constructed for generating a first electrical signal that is indicative of the intensity of at least said first wavelength of radiation, the detector being operatively disposed to responsively intercept the at least one radiation beam exiting the gas flow conduit through the other of the windows;

(e) means for generating a second signal that is indicative of the intensity of radiation of said at least one beam that passes through said analyte gas effectively substantially unattenuated by absorption of radiation by said indicator species; and

(f) signal interpretation means, including electronic data processing means, for analyzing said first and second electrical signals to determine the level of absorption of at least said first wavelength of radiation, by the indicator species, that occurs during passage of said at least one beam through the analyte gas.

16. The apparatus of Claim 15 wherein said first wavelength is in the infrared range, and wherein said means for generating said at least one beam is selected from the group consisting of Fourier Transform Infrared, filter-based, laser spectroscopy, and dispersive spectrometers.

17. The apparatus of Claim 15 additionally including means, operatively disposed in the beam path, for modulating said at least one beam of radiation, such modulation being effective for discriminating radiation of different wavelengths.

18. The apparatus of Claim 15 additionally including a processing chamber for fabrication of silicon semiconductor devices, providing said reaction site.

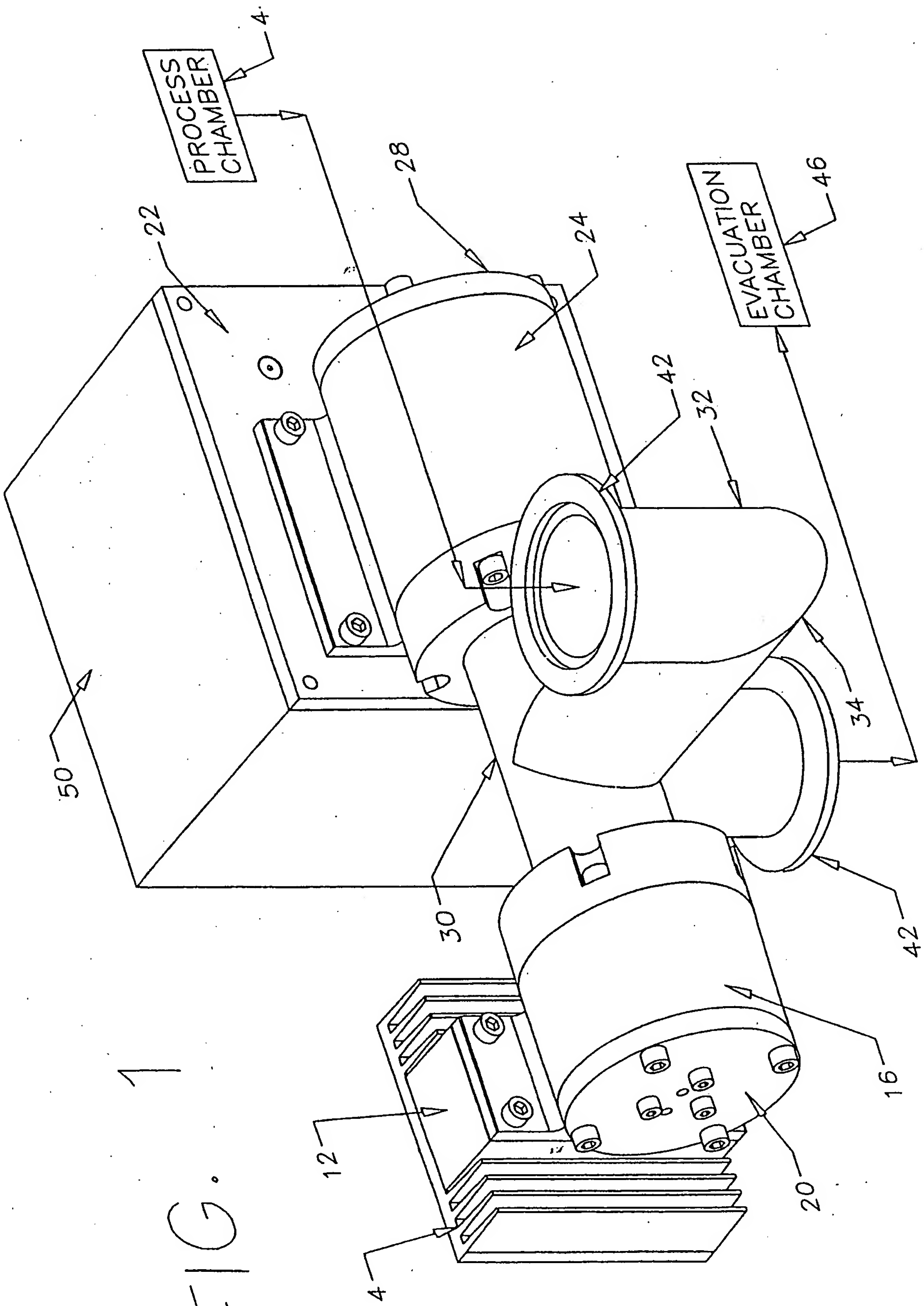

PROCESS CHAMBER
4
28
22
24
EVACUATION CHAMBER
46
42
32
34
50
30
42
16
20
12
14

FIG. 1

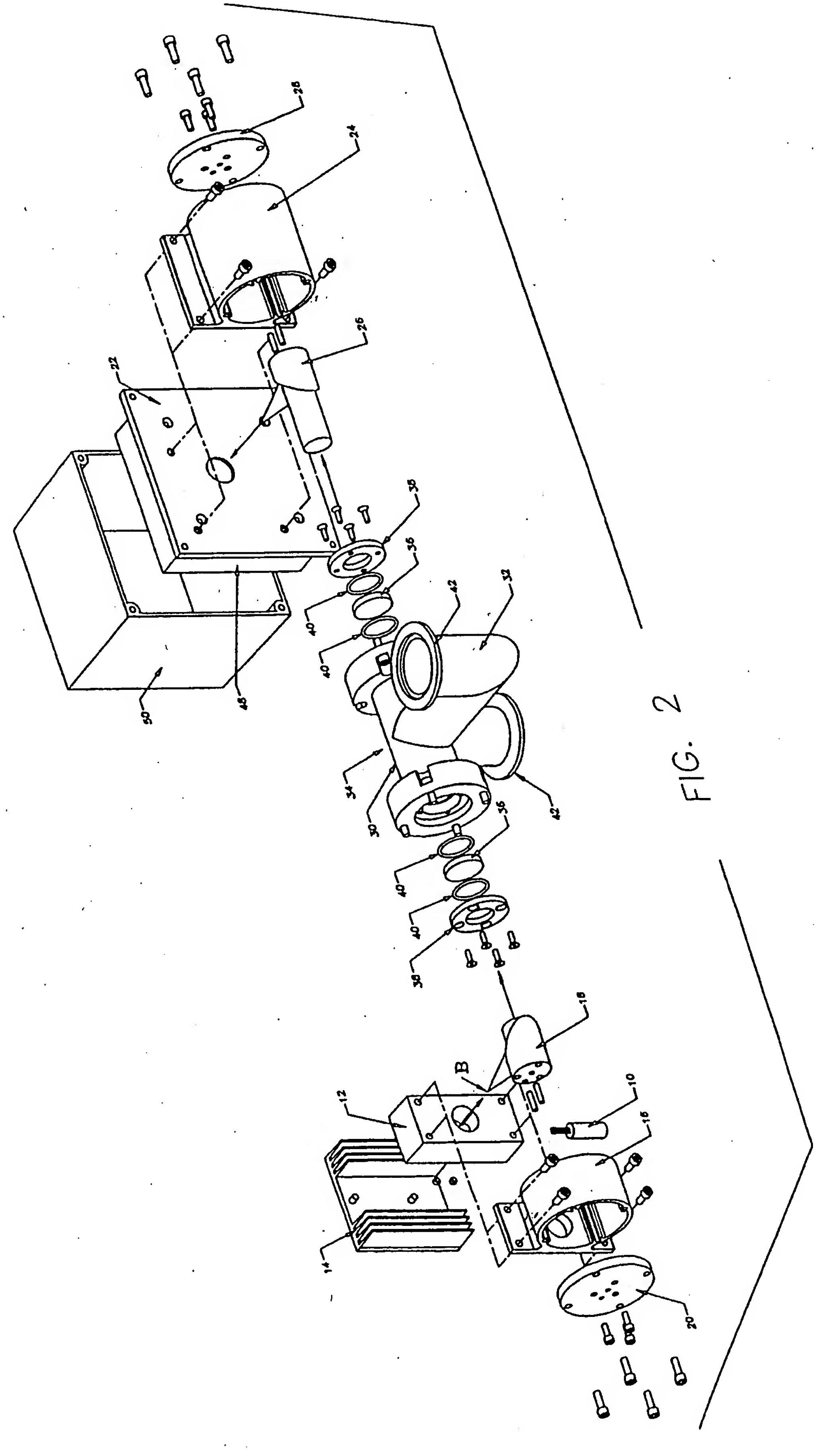

28
24
26
22
38
36
40
40
42
32
50
48
34
30
42
36
40
40
38
18
B
12
10
16
14
20

FIG. 2